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РОССИЙСКОЕ АГЕНТСТВО ПО ПАТЕНТАМ И ТОВАРНЫМ ЗНАКАМ
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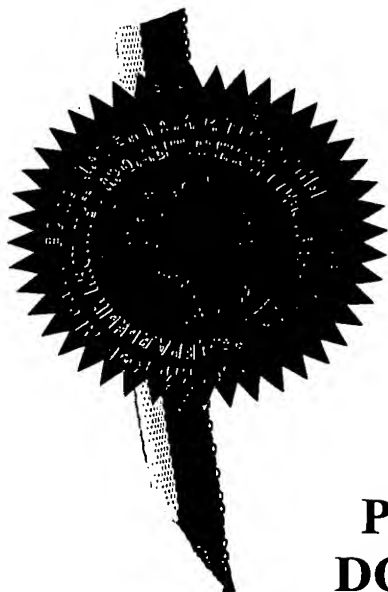
Название изобретения

Method for the generation of low-temperature nitrogen and apparatus therefor

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Title: Method for the generation of low-temperature nitrogen and apparatus therefor

The invention relates to applied chemistry, more specifically to a composition for the generation of gases of low temperature and a process for the obtaining of gases of low temperature.

5 Gas generating processes based on the decomposition or burning of chemical propellants and other compositions are frequently being used for a number of purposes such as the inflation of airbags from, for instance, cars, rafts, life
10 boats and vests, fast installed partitions (which are used in well drifts to cut off the well in case of fire), drives and generators for different types of pneumatic systems and operations mechanisms etc.

Some technical methods for obtaining relative cold gases, in particular nitrogen, are known. These methods are
15 based on the decomposition or the burning of solid materials in special units. These materials are generally shaped in the form of monolithic or porous products and come in all types of shapes and sizes.

The hot gases generated from the decomposition of
20 these materials are in general cooled with the aid of special chemical cooling agents or by specific designed features such as heat exchangers.

The high temperature burning gases are passed through the layer of the cooling agent or the heat exchanger and the
25 temperature of the gases decreases as a result of the endothermal decomposition process of, or heat absorption by the cooling agent. Such processes are described for instance in US-1362349, GB-1371506, FR-136897 and the Russian
inventors certificate 801540. The use of heat exchangers is
30 described in GB-1500137 and GB-1487944.

The degree of cooling of the generated gas depends on the nature of the cooling agent, the mass of the cooling

agent, which can sometimes exceeds the mass of the gas-generating composition, and in case of the heat exchanger, the design features of exchanger.

One of the drawbacks of the prior art as cited above
5 is the relative complicated structure of these units. Another drawback is that the known gas generators do not allow or provide for the gases to be cooled below 150°C. Therefore the applicability of these gas generators is limited to systems that can withstand such high temperatures. This is a
10 disadvantage from a cost-economic viewpoint.

Additionally, gases obtained by the use of the above described methods contain large and undesired amounts of components which may not only have a negative effect on the construction but also in case of airbags for cars, for the
15 person (driver) which is supposed to be protected by the airbag.

Complicated design and complex products resulting in their increasing mass, size and complexity are negative features of these gas generating methods. This decreases
20 reliability and efficiency of the complete system. Especially in the life saving airbags industry there is a continuous need for reliable, safe and economic methods for the generation of cold gases.

RF-patent 2108282 describes a method of generating
25 cold gases, specifically nitrogen, by using the endothermal decomposition of a product made of gas penetrable solid material. The gas penetratable solid material comprises a nitrogen source and a heat absorbing mixture, whereby the gaseous reaction products are cooled by passing the hot gases
30 through the porous body of the product in the moving direction of the reaction front. The hot gases heat the porous body to a temperature necessary to support the endothermic chemical reaction taking place. The heating of the porous body is necessary to enable the main reaction. The
35 decomposition of the cooling agent is also an endothermic chemical reaction. The patent claims to obtain nitrogen gas

from a solid propellant system with a purity of 97-98% and a temperature below 150°C.

One of the disadvantages of the above-mentioned methods is that, when azides are used as the nitrogen source, in general sodiumazide is used for the low temperature nitrogen production. The decomposition reaction of NaN_3 results in Na and N_2 . The formed nitrogen is blown off and the slag remains. This slag comprises of the remains of cementing agent and cooling agent and metallic sodium. Under these conditions of gas generation the highly chemical reactive sodium is thus generated. This highly reactive material will accumulate in the condensed burning products and thus provides a potential hazard for persons involved. When moisture is present this can result in vigorous and dangerous reactions taking place in combination with the generation of the highly flammable and explosive hydrogen. The decomposition of which might be followed by explosions, other undesirable effects or even personal injuries, if persons are involved.

Methods for the neutralisation of sodium are itself known in the art and for instance described in "Sodium production, its properties and use", State Publishing House, Moscow, 1961 pp 142. One of the methods described for the removal of metallic sodium is destruction with water. To be able to apply this method in order to neutralise the used gas generator, the generator after use has to be hermetically sealed and transported to a suitable installation to adequately neutralise the reactive remains of the generator. This is dangerous, cost-ineffective, complex and thus undesirable.

In the case of sodiumazide as the nitrogen source, elemental sodium (Na) is formed upon decomposition of sodiumazide. Sodium is a highly reactive and aggressive chemical. As a result of this reactivity, sodium can react with a wide class of substances to a number of sufficiently stable compounds. One of these compounds is sulphur. Sodium

reacts with sulphur to form sodium sulphide (Na_2S).

The neutralisation of sodium by reaction with sulphur or sulphur compounds in gas generating compositions is known for instance from US 3775199, US 5536340, EP 394103 and US 3741585. The sulphur is vaporised during the decomposition of the gas-generating composition and reacts with the formed sodium slag to the neutral sodiumsulphide.

In the gas generators of the prior art as described hereinabove, the sulphur is vaporised together with the gas generation. It is difficult to vaporise the sulphur at the same rate at which the sodium slag is formed and the rate at which it reacts with the sodium slag. As a result vaporised sulphur will exit the gas generator and/or not all metallic sodium is neutralised. This is a drawback of the use of mixtures of sulphur and gas-generating compositions as described in the prior art.

It is therefore a goal of the present invention to develop a product which will result in the effective generation of nitrogen gas of low temperature without the adverse effects as described above and without major concessions towards output and performance parameters of the gas generator.

It is another goal of the invention to provide for a process for the generation of nitrogen gas of low temperature and to provide for a gas generator which generates nitrogen gas of low temperature.

Inventors have now found a gas-generating configuration that can overcome the above-mentioned deficiencies of the prior art and result in the generation of low temperature nitrogen gas with effective and sufficient neutralisation of the reactive slag.

The invention accordingly comprises a gas generator comprising a first body, comprising means for the generation of gas, and a second body, comprising means for the generation of a neutralisation agent, wherein means are present for contacting the neutralisation agent with the

reaction products formed in the generation of gas in the first body, and wherein means are present for operating the means in the second body at a temporal and/or spatial interval with the means in the first body.

5 The principle encompasses two gas generators in one housing. A first gas generator with the primary task of generating gas, preferably of low temperature, and a second gas generator with the primary task of generating neutralising compounds for the slag obtained from the first gas generator.

10 The first gas generator contains a composition from which nitrogen gas, preferably of low temperature can be obtained by the decomposition of a gas generating composition in the form of a gas penetrable solid material wherein the generated gaseous products are passed through the porous body in the moving direction of the reaction front.

15 The second gas generator (the neutraliser) is another composition, comprising a gas generating composition together with an effective neutraliser compound, for instance sulphur. 20 With the neutraliser composition gas and vaporised sulphur is generated at a time and space interval with the first gas generator. The gas and vaporised sulphur is generated at a rate and a manner that the effective neutralisation of slag is accomplished and the vaporised sulphur is not emitted. The 25 vaporised sulphur reacts with the reaction products from the first gas generator such that the products are effectively neutralised.

30 In an embodiment the invention thus relates to a first gas generator comprising a gas penetrable solid material comprising a nitrogen source, preferably an azide, more preferably sodiumazide, cementing agent and optionally a heat absorbing mixture, wherein the solid material has a porosity of 35-60 % and a second gas generator containing a neutraliser composition which contains sulphur and an 35 additional nitrogen source.

 In an embodiment of the invention the nitrogen

sources in both the first and the second gas generator are selected from the group of alkalimetal azides or an earth-alkalimetal azides, preferably potassium azide or sodium azide, more preferably sodium azide.

5 A typical embodiment of the invention is as follows.

A body consists essentially of two parts: the gas generator and the neutraliser. The gas generator will contain a porous solid material, containing a gas generating component such as sodiumazide, together with cementing agents
10 and optionally cooling agents or other heat absorbing mixtures. The other part of the body is the neutraliser mass. The neutraliser contains the neutraliser (sulphur) and a gas generating component. The gas-generating component may be identical to the gas generating component in the first part,
15 for instance sodiumazide. When the gas generator is activated, gas is generated and blown off, leaving behind highly reactive metallic sodium slag. The neutraliser is activated and the sulphur is vaporised. The vaporised sulphur will react with the sodium, resulting in the neutral
20 sodiumsulphide.

The amount of sulphur is such that it is sufficient to effectively neutralise the slag formed in both the neutraliser and the gas generator and that only minimal or almost no vaporised sulphur is blown off.

25 In the present invention, in order to facilitate the interaction between the sodium and the neutraliser compound (e.g. sulphur) it is preferred that the neutralisation product is in a form in which the reaction with the sodium slag is enhanced. To this extent the sulphur in the
30 neutraliser can be mixed with the gas-generating compound in the form of powder, granules, etc.

In a gas generator according a preferred embodiment of the invention, the combined amounts of the nitrogen sources in the first and second body comprises 50-80 wt.%
35 drawn on the total weight of the gas generator and the amount of neutralisation agent in the second body 47-90 wt.% of

neutralisation agent, drawn on the weight of the second body. The respective weight of the gas generator is measured in the absence of housing, external cooling aids, etc.

The second gas generator comprises between 17 and 35 wt.% of the gas generator according to the invention, drawn on the total weight of the gas generator. The second gas generator contains 10 to 53 wt.% of the nitrogen source and 47 to 90 wt.% of neutralising agent. In a preferred embodiment the second gas generator contains 15 to 25 wt.%, more preferable 17 to 23 wt.% of nitrogen source and 75 to 85 wt.%, more preferable 77 to 83 wt.% of sulphur.

In a preferred embodiment the sulphur is in a particulate form, preferably in the form of small particles, more preferably in the form of sulphur powder.

The first and second gas generator do not have to be physically separated from each other. In embodiments of the invention they can be placed in any position relative to each other, as long as the vaporised sulphur of the second generator can come into contact with the slag from the first generator.

In an embodiment of the invention, the reaction between sodium and sulphur takes place behind the reaction front of the decomposition reaction of the first gas generator. The spatial interval between the reaction front of the first gas generator and the second gas generator is placed so that the reaction products of high temperature from the first gas generator stay behind, while the optionally cooled nitrogen gas is blown off.

In another embodiment of the invention the rate at which the gas generating composition decomposes is different from the decomposition rate of the neutraliser charge. Thus, the decomposition of the gas generating composition and the neutraliser are started simultaneously. Metallic slag is formed, followed by the generation of vaporised sulphur which neutralises the sodium.

In another embodiment of the invention the time at

which the neutraliser is activated is later than the activation time of the gas generator.

The relative amounts of sodium azide and sulphur are contained between the lower limit of sulphur which is the amount of sulphur necessary for the neutralisation of the elemental sodium formed. The upper limit of sulphur is determined by the amount at which almost no vaporised sulphur will be blown off or the amount which is considered acceptable with respect to output gas purity.

The rate at which the gas was generated was determined in order to provide for an optimal formulation together with the optional heat absorbing product and the neutraliser product. The ratio of the different components (nitrogen source, heat absorbing material and sulphur) was chosen such that the required maximum discharge of vaporised sulphur and the stable burning of the material was obtained. It was found that a stable ignition and burning of the material was not possible if the concentration of the sulphur in the material was more than 90 wt.% of the combined weight of additional nitrogen source and sulphur (neutraliser mass). If the concentration of sulphur was below 47 wt.% of said combined weight, the discharge of vaporised sulphur decreased below the desired level and the total (neutraliser mass)/(nitrogen source) ratio had to be increased in order to obtain the bonding of the elemental sodium in sufficiently high levels. The preferred mass ratio of the nitrogen source and the neutraliser is determined by the total neutralisation of sodium to sodium sulphide in the slag.

In a preferred embodiment of the invention the nitrogen source and the neutraliser, preferably sulphur, are homogeneously mixed.

In another preferred embodiment of the invention, the neutraliser product comprises sulphur and additional nitrogen source in an amount of 10-53 wt.% of the additional nitrogen source and 47-90 wt.% of sulphur, based on the combined weight thereof.

In this embodiment of the invention, the combined amount of the nitrogen source and sulphur, based on the total weight of the product is from 17 to 35 wt.%.

5 In case the combined amount of the additional nitrogen source and the sulphur is less than 17 wt.%, the total neutralisation of sodium is insufficient because of lack of sulphur. In case the amount is above 35 wt.%, the vaporised sulphur will be blown off with the generated gas and thus the purity of the generated nitrogen gas decreases.

10 The invention also relates to a process for the generation of low temperature gases, preferably nitrogen, comprising the steps of:

- exothermic decomposition of a gas-penetrable porous solid material in a first body;
- 15 - decomposing a solid in a second body containing a gas generating solid and a neutralisation agent;
- neutralising the generated reaction products from the first body by reaction with the neutralisation agent;
- 20 - maintaining a temporal and spatial interval between a decomposition front of the first body and a decomposition front of the second body.

25 Upon ignition of the nitrogen source containing material and the neutralisation material, the materials start burning. The gaseous burning products of the nitrogen source pass through the ramified porous body in the moving direction of the reaction front and are cooled by transferring heat to the porous body. At the burning of the neutraliser, vaporised sulphur is generated and passed through the slag of the
30 nitrogen source. In an embodiment of the invention a spatial and temporal interval between the reaction front of the nitrogen source and the reaction front of the neutraliser is provided. The reaction between the vaporised sulphur and the
35 metallic sodium is exothermic. However, as there is a spatial and/or temporal interval between the gas generation and the

neutralisation, this will not influence the temperature of the generated gas. This interval can be accomplished by a lower reaction rate of the neutraliser when compared to the reaction rate of the nitrogen source. By this interval the vaporised sulphur is mainly generated after the sodium is formed, thus allowing for more optimal reaction conditions for both the generation of gas and the neutralisation of sodium.

The interval can also be controlled by design related features such as the adjustment of the flow rates by a different form of the burning surface or by the non-simultaneous ignition of the nitrogen source and the neutraliser. The invention accordingly comprises a generator for low temperature gas.

In an preferred embodiment the generated gases are cooled by passing the gases through a porous body in the moving direction of the reaction front.

In an preferred embodiment heat is absorbed which is formed in the exothermic reaction by a heat absorbing material included in the porous body.

In a preferred embodiment of the invention the amount of heat generated in relation to the amount absorbed heat is such that the generated gas is cooled to a temperature below 150°C, preferably below 100°C.

The invention also encompasses a gas generator for obtaining low temperature nitrogen gas comprising means for (in)dependent ignition of the first and second gas generator, means for generating nitrogen gas by the decomposition of sodium azide, means for cooling the nitrogen gas and means for neutralising the sodium and means for venting the nitrogen gas.

Claims

1. Gas generator comprising a first body, comprising means for the generation of gas, and a second body, comprising means for the generation of a neutralisation agent, wherein means are present for contacting the
5 neutralisation agent with reaction products formed in the generation of gas in the first body, and wherein means are present for operating the means in the second body at a temporal and/or spatial interval with the means in the first body.
- 10 2. Gas generator according to claim 1, wherein the means in the first body comprise a gas-penetrable solid material comprising a nitrogen source, preferably an azide, more preferably sodium azide, cementing agent and optionally a heat absorbing mixture, wherein the solid material has a
15 porosity of 35-60 wt.%.
3. Gas generator according to claim 1 or 2, wherein the reaction products comprise slag containing sodium.
4. Gas generator according to any of the claims 1-3, wherein the second body contains a nitrogen source and a
20 neutralising agent.
5. Gas generator according to any of the claims 1-4, wherein the neutralisation agent is vaporised sulphur.
6. Gas generator according to any of the claims 1-5, wherein the combined amounts of the nitrogen sources in the
25 first and second body comprises 50-80 wt.% drawn on the total weight of the gas generator and the amount of neutralisation agent in the second body 47-90 wt.% of neutralisation agent, drawn on the weight of the second body.
7. Gas generator according to any of the claims 1-6,
30 ~~wherein the second body is between 17 and 35 wt.%, drawn on the total weight of the gas generator.~~
8. Gas generator according to any of the claims 1-7, wherein the second body contains 10 to 53 wt.% of the

nitrogen source and 47 to 90 wt.% of the neutralisation agent.

9. Gas generator according to any of the claims 1-8, wherein the generated gases are cooled by a heat absorbing material.

10. Gas generator according to any of the claims 1-9, whereby the heat absorbing material is included in the first body.

11. Process for the generation of gases, preferably nitrogen, comprising the steps of:

- exothermic decomposition of a gas-penetrable porous solid material in a first body;
- decomposing a solid in a second body containing a gas generating solid and a neutralisation agent;
- neutralising the generated reaction products from the first body by reaction with the neutralisation agent;
- maintaining a temporal and spatial interval between a decomposition front of the first body and a decomposition front of the second body.

12. Process according to claim 11, wherein the generated gases are cooled by passing the gases through a porous body in the same direction the reaction front is moving.

13. Process according to claims 11 or 12, wherein heat is absorbed which is formed in the exothermic reaction by a heat absorbing material included in the porous body.

14. Process according to claims 11-13, wherein the amounts of heat are such that the generated gas is cooled to a temperature below 150°C, preferably 100°C.

13. Gas generator for obtaining low temperature nitrogen gas comprising means for the dependent or independent ignition of the first and second gas generator, means for generating nitrogen gas by the decomposition of sodium azide, means for cooling the nitrogen gas and means for neutralising the sodium and means for venting the nitrogen gas.

Abstract

Gas generator comprising a first body, comprising means for the generation of gas, and a second body, comprising means for the generation of a neutralisation agent, wherein means are present for contacting the neutralisation agent with the reaction products formed in the generation of gas in the first body, and wherein means are present for operating the means in the second body at a temporal and/or spatial interval with the means in the first body.